Particle Imaging Velocimetry of Self-Assembled Monolayer Formation

Abstract

Small surface functionalized particles can be made to form densely packed two dimensional layers on a water-air interface in a process known as fluid forming. Using a laser-based particle imaging velocimetry (PIV) system, the formation of these monolayers can be observed in real time. This report documents the synthesis and preliminary image analysis of particles suited for these investigations, establishing a proof of concept for future experiments.

Introduction

Silica particles are an ideal candidate for this study. Using a modified Stöber synthesis method,[1] highly monodisperse silica particles in the range of 300 nm to 1 μm can be easily produced. These particles may then be coated with organosilanes with long carbon chains (such as octadecyltriethoxysilane) to give them a hydrophobic surface character. These surface functionalized particles will closely pack on a water-air interface in order to minimize contact with the underlying water.

Silica Particle Synthesis

The synthesis procedure used to produce monodisperse silica spheres was adopted from a 2007 paper by M. Shishido and D. Kitagawa[2], which itself was based on the Stöber synthesis method. All chemicals used were supplied by Fischer scientific. 26.5 mL of 29 wt. % ammonium hydroxide solution was combined with 17.2 mL of deionized water and 189.7 mL of reagent grade (95 wt. %) ethanol and briefly stirred in a 500 mL beaker. 16.6 mL of tetraethylorthosilicate (TEOS) was then added to beaker, which was then covered with a layer of Parafilm. The contents were stirred for 2 hours at room temperature with a magnetic stirring bar. The liquid containing synthesized particles was then centrifuged for 10 minutes at 15,000 RPM. After draining the supernatant and dispersing the caked particles in ethanol, the contents were again centrifuged. This process was repeated a total of three times, yielding silica particles dispersed in ethanol.

Characterization of Synthesized Silica Particles

Dynamic light scattering (DLS) with a Coulter LS 230 was used for rough characterization of the particles. The particles were diluted in deionized water before scattering. The DLS analysis showed a mean particle size of 468 nm, but a high coefficient of variation of roughly 249%. **Figure 1** shows the results of the dynamic light scattering analysis. Note the symmetrical lognormal distribution. This suggests that the normal distribution is skewed to the right, which in turn suggests that agglomeration may be significant. However, the particles were additionally characterized by scanning electron microscopy (SEM) to get more accurate coefficient of variation and level of agglomeration estimates. **Figure 2** shows an SEM image of the dried particles. The diameters of 20 randomly chosen particles in the image were measured by hand. According to this analysis, the mean particle size is 457 nm and the coefficient of variation is only 3.4%. In addition, the level of agglomeration seems acceptably low in the micrograph, especially since this particular sample was not sonicated before the image was taken.

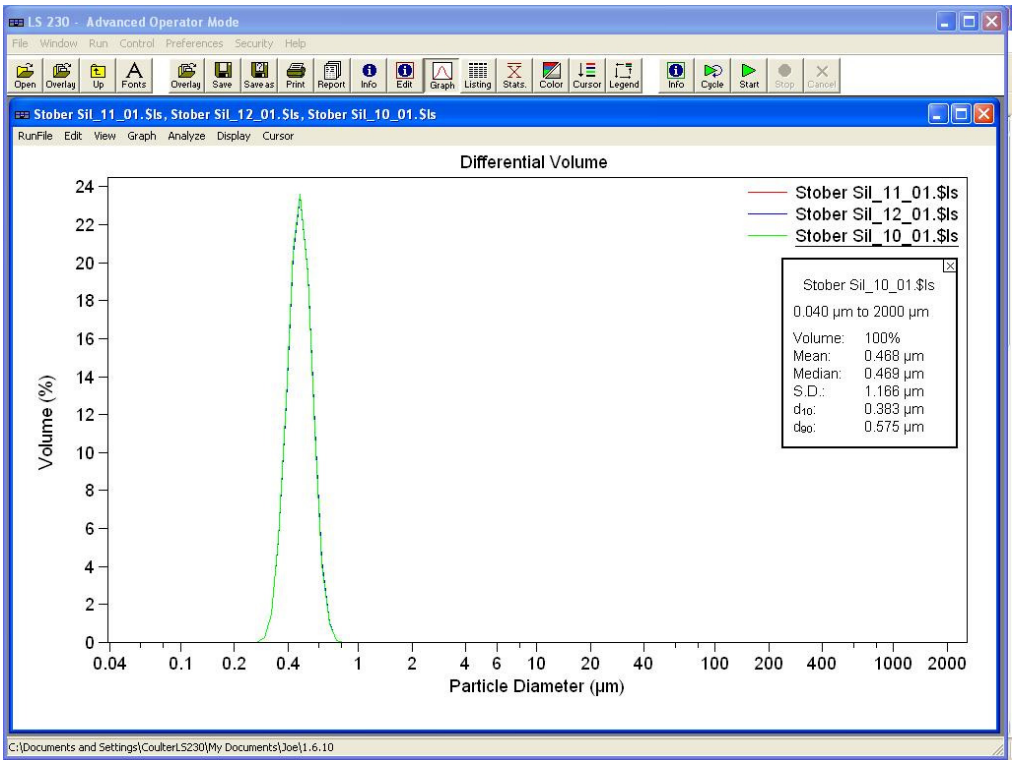

Figure 1 - dynamic light scattering data for synthesized silica particles

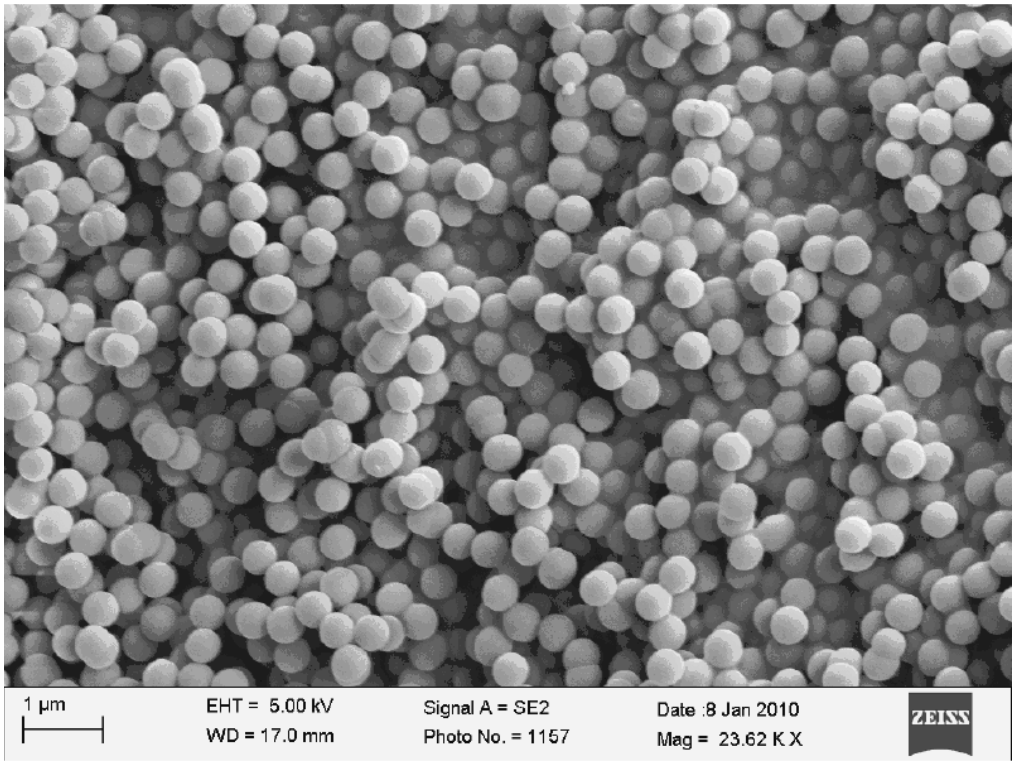

Figure 2 - SEM micrograph of synthesized silica particles

Surface Functionalization of Synthesized Silica Particles

The functionalization method employed conditions found by Hyun Jun Kim and used on 11/8/07[3]. 0.1 g of the synthesized silica particles were mixed with 10 mL of ethanol. This was added to 0.4 mL of a solution comprising 0.8 mL octadecyltriethoxysilane and 40 mL ethanol. The mixture was stirred overnight at room temperature with a magnetic stirring bar in a 50 mL beaker. During this time, ethanol was released as the octadecyltriethoxysilane molecules reacted with the silica, affixing many long carbon chains to the surfaces, making them hydrophobic. The reacted particles were centrifuged and washed with isopropanol for 10 minutes at 15,000 RPM three times. 10 mL of 10 mg/mL silica in isopropanol was prepared as a base mixture to be further diluted when experimenting with film preparation. Note: octyltriethoxysilane, as well as other long-chain organosilanes, may also be used to functionalize the particles, though the shorter chain of carbon atoms imparts a lesser degree of hydrophobicity to the particles, which may significantly decrease the 2D packing factor. The investigation of 2D packing factor vs. functionalizing agent is another task yet to completed within the scope of this project.

Basic Film-Making Procedure

All subsequent experiments involved spreading the functionalized silica particles in isopropanol over a water-air interface with controlled area at a specified flow rate. This was achieved using a Langmuir-Blodgett trough as shown in **Figure 3**. It is comprised of a 1 L beaker filled with deionized water and a top-mounted apparatus that controls the area of the water surface. A glass slide attached to a controlled vertically oriented continuous motor was inserted vertically into the water adjacent to the Teflon separator in the center of the apparatus. The motor controller was a Newport Univeral Motion Controller Model ESP300. Functionalized silica particles in isopropanol were loaded into a B/D 20 mL syringe which itself was loaded into a Baxter Model AS40A Infusion Pump capable of controlled flow rates between 1 and 120 mL/hr.

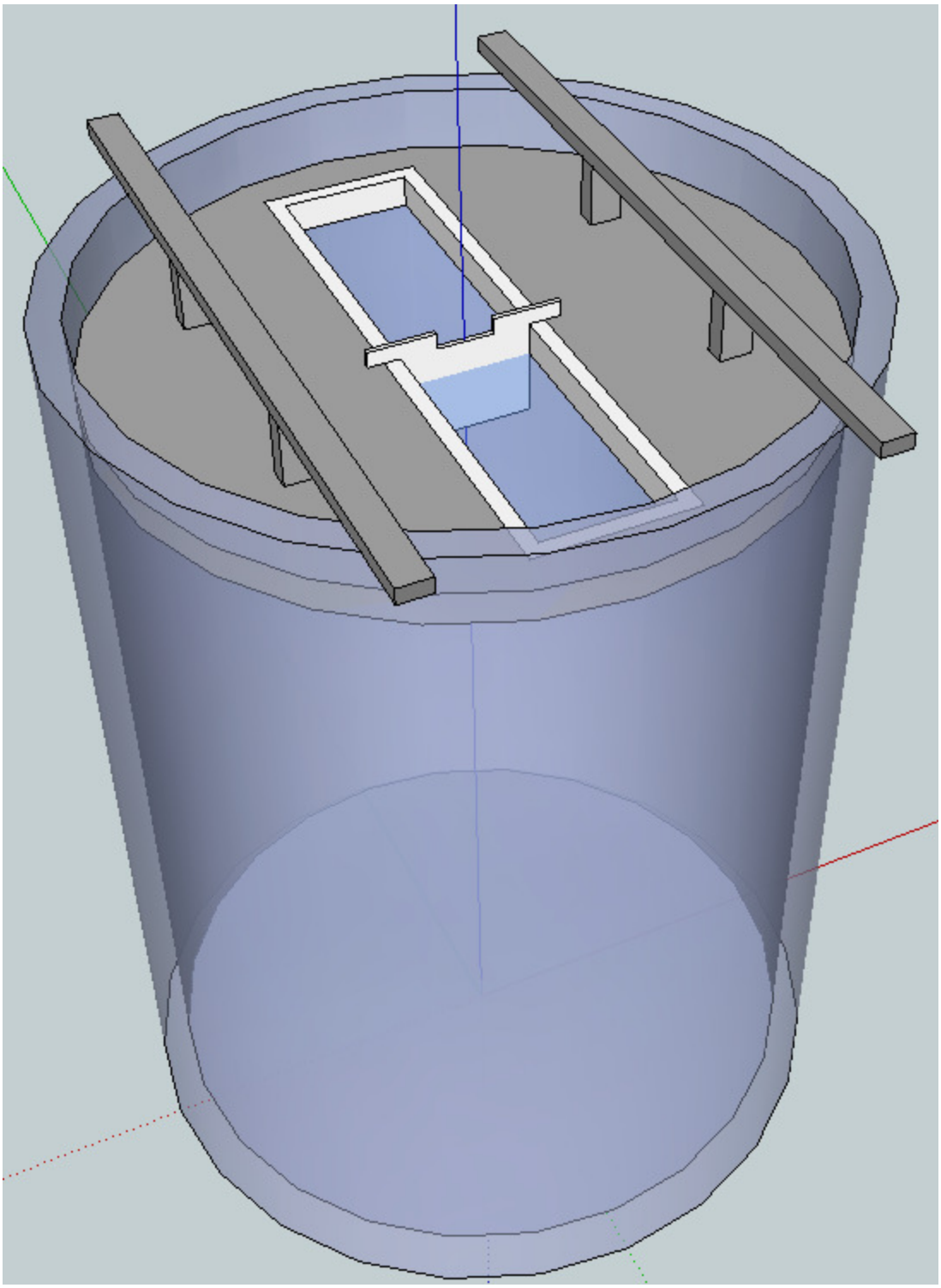

Figure 3 - the Langmuir-Blodgett trough used throughout the film-making experiments (sketch made using Google SketchUp)

The syringe needle was bent downwards so as to just touch the top of the trough, allowing the particles in isopropanol to smoothly spread onto the water-air interface and minimally disturb the existing particle arrangement.

To estimate a good initial withdrawal rate, a mass balance calculation was carried out to equalize the area withdrawal rate with the coverage rate. The final equation is $R_w = \frac{(f_v)(R_f)}{480(W_s)(PF)(r_p)}$, where R_w = withdrawal rate in mm/s, f_v = volume fraction of particles in the liquid, R_f = feed rate in mL/hr, W_s = slide width in mm, PF = 2D particle packing factor, and r_p = particle radius in cm. For all of the experiments carried out in this report, PF was assumed to be 0.9069 and r_p was 229 nm, or 0.00029 cm.

Finding Ideal Film-Making Conditions

For the initial experiments, the silica particles in isopropanol were diluted to 10 mg/mL, roughly equivalent to 0.04 % by volume. In the first few films made, imperfections on the glass slide surface were limiting the overall film quality. As such, a batch of slides were soaked in 1M nitric acid for 2 hours, and subsequently rinsed and stored in deionized water, only to be taken out immediately before use. At this point, it was also clear that at some times the withdrawal rate had to be adjusted on the fly - the calculated withdrawal rate could be used initially, adjustments are necessary once the withdrawal has begun due to slight fluctuations with each experiment (such as slide positioning in the trough). As such, the data collected notes both initial and final withdrawal rates. The experiments shown in **Table 1** below, were performed on 1/15/10.

Sample ID	mg particles per mL liquid	Vol. % Particles	Feed Rate (mL/hr)	Initial Withdrawal Rate (mm/s)	Final Withdrawal Rate (mm/s)	Comment
PIV_1_1801	1.0	0.038	10	0.153	0.140	Excellent
PIV_1_1802	1.0	0.038	20	0.305	0.300	Good
PIV_1_1803	1.0	0.038	30	0.458	0.450	Fair
PIV_1_1804	0.5	0.019	10	0.076	0.076	Poor
PIV_1_1805	0.5	0.019	20	0.153	0.153	Poor
PIV_1_1806	0.5	0.019	30	0.229	Faster needed	Very Poor
PIV_1_1807	1.5	0.057	10	0.229	0.229	Good
PIV_1_1808	1.5	0.057	20	0.458	Slower needed	Poor
PIV_1_1809	1.5	0.057	30	0.687	0.687	Poor
PIV_1_1810	1.5	0.057	10	0.229	0.229	Fair
PIV_1_1811	1.0	0.038	10	0.153	0.153	Fair
PIV_1_1812	1.0	0.038	20	0.305	0.305	Very Poor
PIV_1_1813	1.0	0.038	30	0.458	0.458	Very Poor

Table 1 – qualitative assessment of film quality vs. volume % particles and feed/withdrawal rate

The "comment" section is a rough estimate of the film quality based on visual inspection. The difference between a quality film and a subpar film is quite obvious. A quality film will appear smooth and uniform, and also exhibit some diffraction of light, making the film appear red or blue at different angles. Conversely, a poor film will appear blotchy, exhibit uneven diffraction, and may contain striations. These striations are the result of the withdrawal rate being too small, causing the particles to build up too quickly on the water-air interface and disrupting the arrangement at the addition point. In any case, the experiments above show that the conditions that give the most reproducibly good films are 0.038 vol. % particles with a 10 mL/hr feed rate.

Particle Imaging Velocimetry Setup

It should be noted that while 468 nm particles are easily synthesized and self-assembled, they are quite small (on the order of the wavelength of light) and therefore very difficult to observe using conventional imaging techniques. Optical methods are the only option for performing particle imaging velocimetry experiments without requiring an extremely expensive and specialized experimental setup.

As such, in order to resolve individual particles and produce more meaningful PIV data, 10 µm plain silica particles were purchased from Corpuscular, Inc. and functionalized in the same way as the synthesized particles (monodisperse silica particles greater than 1 µm are quite difficult to synthesize). Due to their larger size, these particles do not self-assemble as reliably as their smaller counterparts, but this was a necessary concession to establish a proof of concept for the PIV experiments, as velocity fields and individual particle velocities cannot be calculated using sub-micron particles.

The diagrams below show the physical setup created to perform PIV experiments, which was located in D-107 in the engineering building on Busch campus, a laboratory under the supervision of Professor Tse in the Department of Mechanical and Aerospace Engineering. The required components include:

- Tempest and Gemini 532 nm Nd-YAG dual head laser[4]
- Redlake MASD Inc./Kodak MegaPlus Model ES 1.0 Series Camera[5]
- (2) 532 nm specialized mounted mirror
- ~4 mm focal length cylindrical lens
- ~4 mm focal length convex lens
- 1L beaker w/ Langmuir trough
- Baxter Model AS40A Infusion Pump
- Adjustable stand for infusion pump
- B/D 20 mL syringe
- Any precision multi-channel (at least 3) pulse generator
- Any computer with EPIX XCAP[6] software and a serial connection port

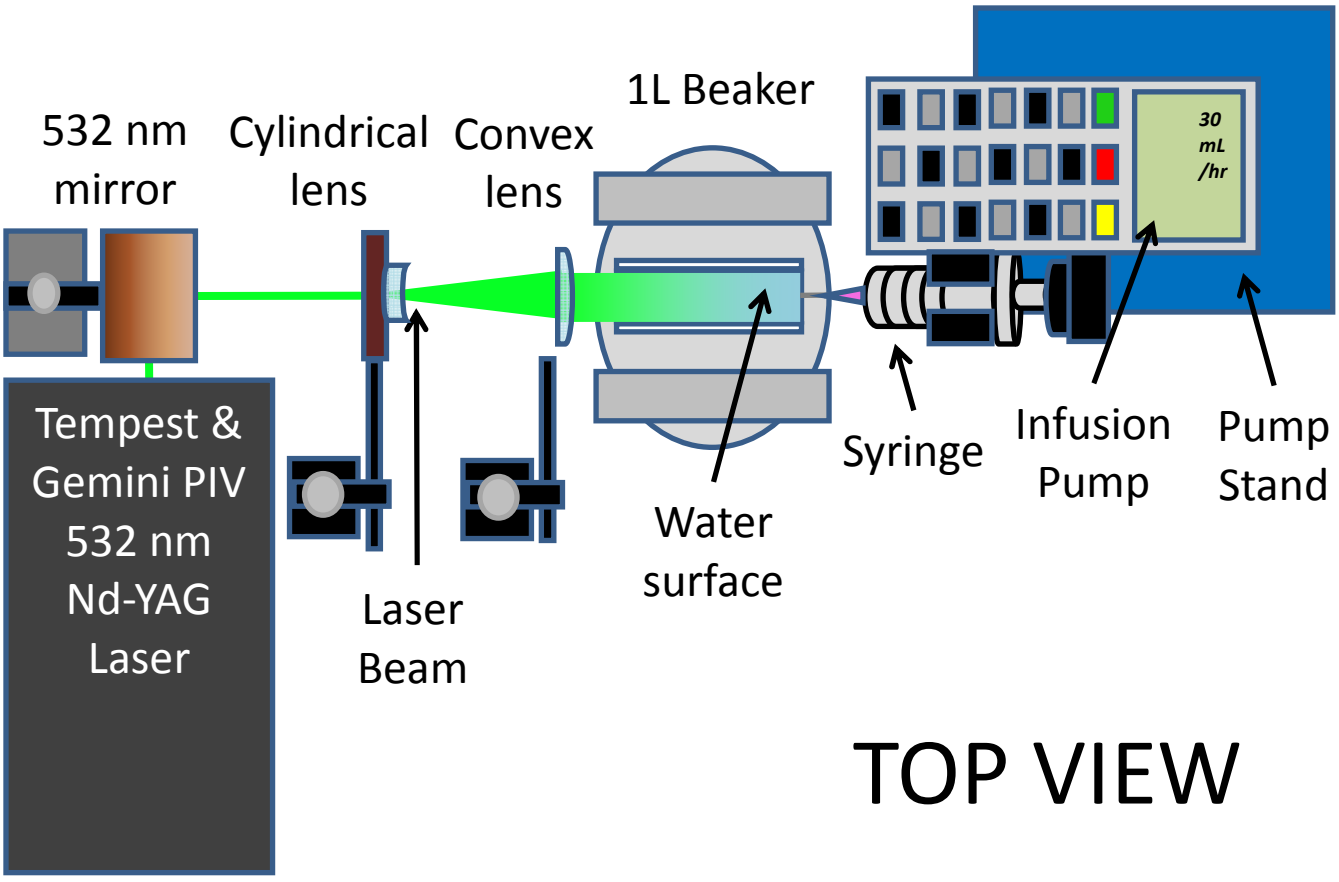

Figure 4 – Top view of laser/camera PIV setup

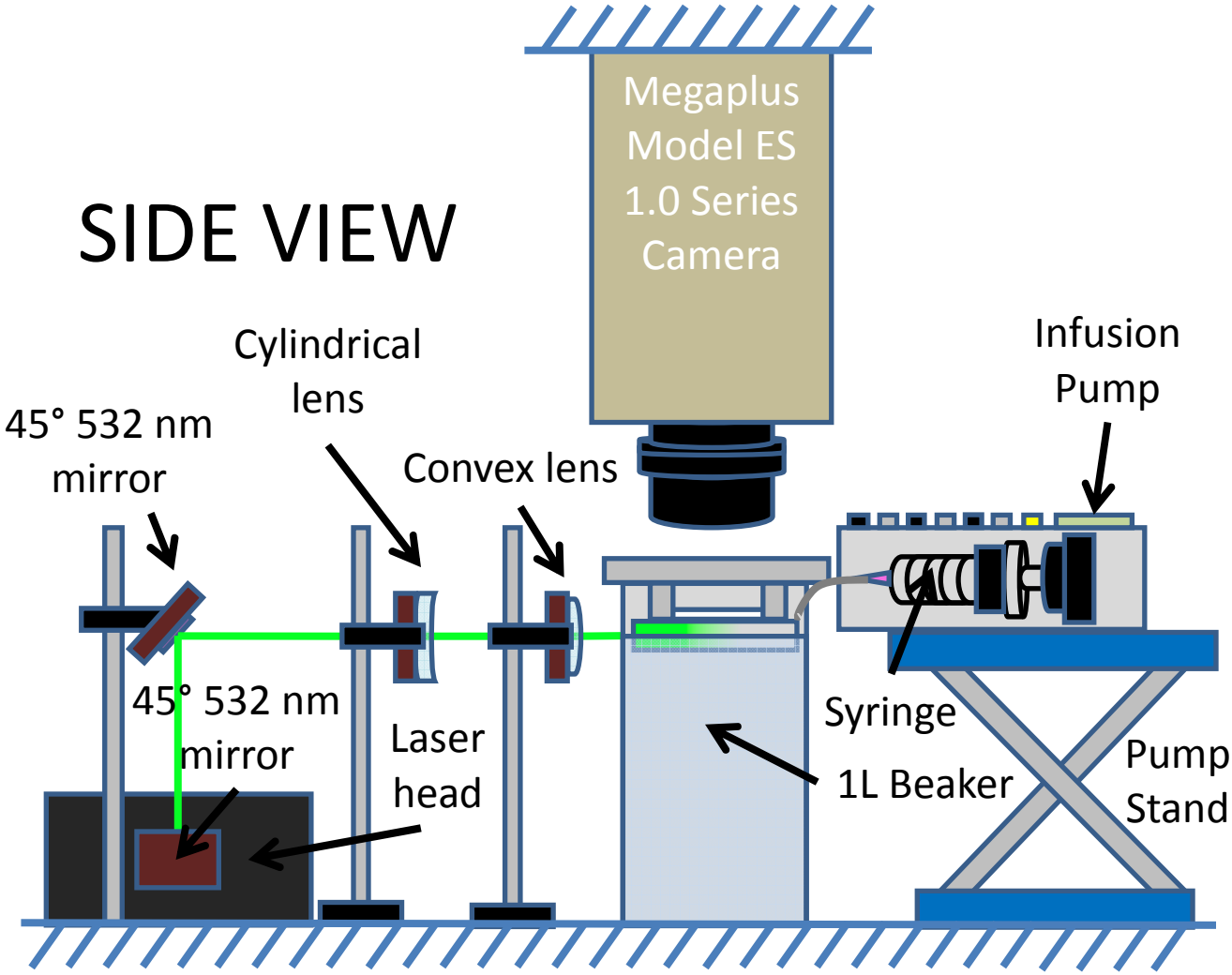

Figure 5 – side view of laser/camera PIV setup

In the above setup, two laser pulses are fired in quick succession while particles are moving on the water surface enclosed by the Langmuir-Blodgett trough. The particles are fed at a controlled rate onto the water surface by an infusion pump that slowly compresses the plunger of a syringe filled with a particular volume percentage of particles suspended in isopropanol, in a similar manner to the film-making experiments described above. A multi-channel pulse generator is used to time these pulses, which are also synchronized with two separate exposures of a camera situated directly above the water surface. The laser pulses are reflected upwards and then to the right by two consecutive 45° mounted 532 nm Nd-YAG mirrors. The laser beams are then spread horizontally by a cylindrical lens, and in turn made parallel again by a convex lens of similar focal length. The distance between the two lenses should be arranged such that the final widened parallel beam has roughly the same width as the short dimension of the trough. The beam, which is vertically aligned with the plane of the water surface, then passes through the glass of the beaker and illuminates any particles situated on the surface, while the camera captures two images of the illuminated particles in quick succession and sends them to a computer running XCAP software (or any other compatible PIV image-capturing software) via serial RS-232 connection.

Triggering and Timing

The multi-channel pulse generator must be preconfigured with appropriate timings to properly conduct PIV experiments. These timings are dependent on the internal delays in both the lasers and the camera. The following diagrams illustrate the timing of these components in the configurations used throughout the described PIV experiments.

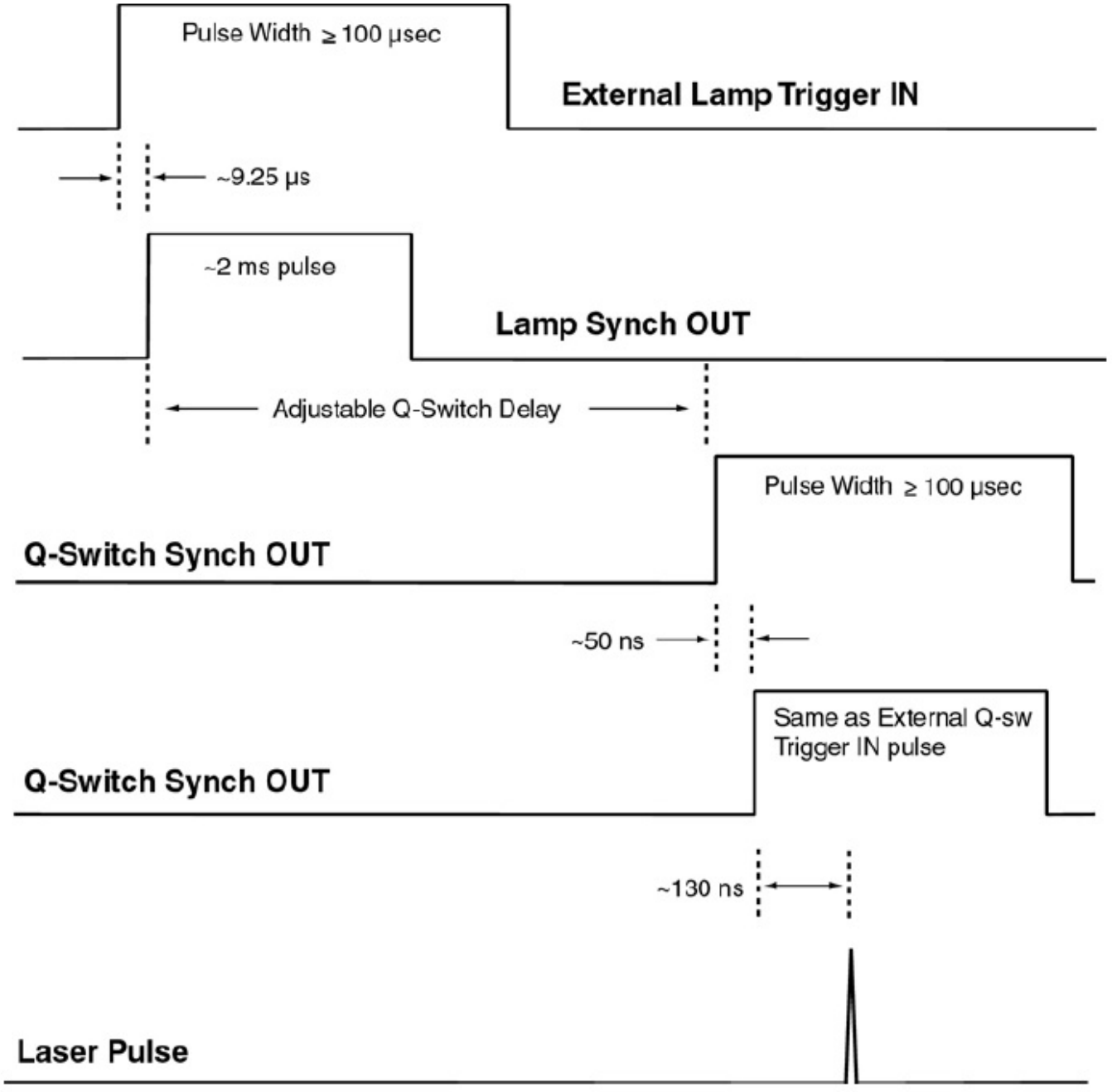

Figure 6 – timing diagram for laser using external triggering[4]

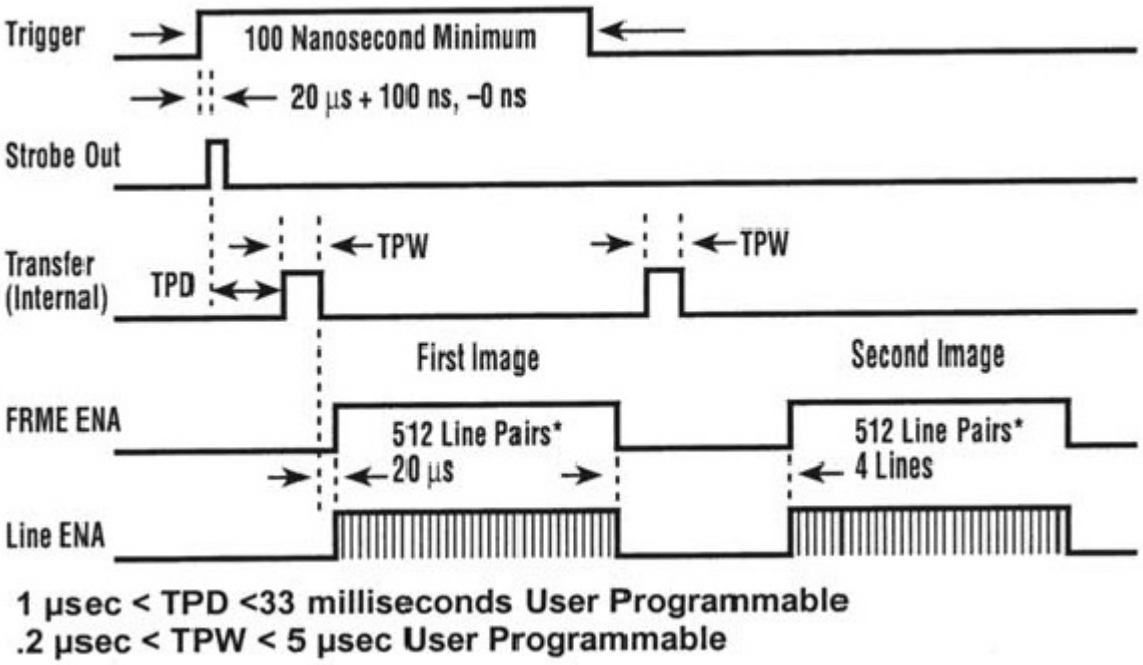

Figure 7 – timing diagram for camera on double exposure mode[5]

In the laser timing diagram, the default Q-switch delay in the laser is 200 µs. In the camera timing diagram, "TPD" refers to the transfer pulse delay, which is adjustable, and "TPW" refers to the transfer pulse width, which is by default 5.0 µs. The transfer pulse delay can be set to equal the camera exposure, which can be adjusted from 1 µs to 33 ms. To properly capture images, the first laser pulse must occur during the first camera exposure, and the second laser pulse must occur during the second camera exposure. In the experiments conducted, the exposure time was maximized to 33 ms to create a bright image. The transfer pulse delay was also set to equal the camera exposure time, and the transfer pulse width was left at the default of 5.0 µs. As such, the first exposure started roughly 33.005 ms after the trigger pulse and ended 66.005 ms after the trigger pulse, and the second exposure started roughly 66.01 ms after the trigger pulse and ended 99.01 ms after the trigger pulse. Since the delay between a trigger pulse to the laser is short (slightly more than 200 µs), the trigger pulses for the lasers were set to occur roughly halfway into the camera exposures.

Three channels were necessary on the pulse generator to create the trigger pulses required for these experiments. The first channel corresponded to the first laser pulse, the second to the second laser pulse, and the third to the camera exposure. The following table shows the timing for the three channels. The pulse widths were chosen to be well over the minimum time.

Channel #	Pulse Delay (ms)	Pulse Width (ms)
1 – first laser pulse	49.5	1.0
2 – second laser pulse	82.5	1.0
3 – camera exposure	0.00	1.0

Table 2 – proper channel timings for pulse generator.

Particle Imaging Velocimetry Procedure

1. 532 nm Nd-YAG laser initialization.
    a. Turn on both laser power supplies by turning keys.
    b. Set the flashlamp setting on both power supplies to external triggering.
    c. Set the Q-switch setting on both power supplies to internal triggering.
    d. Set both lasers to standby mode using the remote control boards.
    e. Use the remote control boards to fire the lasers briefly, and adjust both lasers' power levels to bring the brightness to a suitable, uniform level. If the lasers make a loud click with each firing, the power level is probably set too high.
    f. Always operate within safe power levels and use appropriate eye protection!
    g. Return both lasers to standby mode when finished adjusting the brightness.
2. Particle preparation.
    a. Load the particle-containing liquid into the syringe (~10 mL is usually enough).
    b. Attach a clean, bent needle to the end of the syringe.
    c. Load the syringe into the infusion pump.
    d. Select the proper settings on the infusion pump, and choose an appropriate feed rate in mL/hr (the described experiments were performed at 30 mL/hr.
    e. Adjust the height of the infusion pump so that the needle tip just touches the top of the trough on the beaker. The liquid needs to flow smoothly onto the water surface.
3. Software preparation.
    a. Start the EPIX XCAP software on the computer.
    b. If the camera is connected properly, the following window should appear:

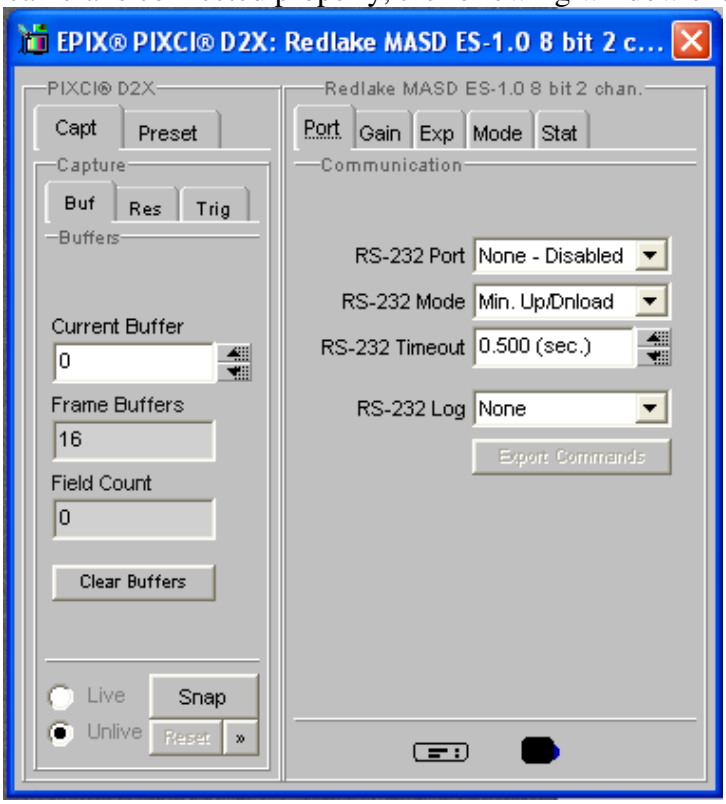

Figure 8 - The XCAP Buffer Screen

XCAP works with an image buffer system. Each time a pair of images is taken, the current buffer and the buffer immediately following it will store the images. Up to 8 pairs of images may be stored at a time.

c. Click the "Live" radio button to see a continuous image from the camera. Use this mode to focus the camera – ideally, an object such as a ruler can be used to focus in the same plane that will be investigated, as this will also give an indication of the level of magnification to create a scale.
d. Once focused, click the "Unlive" radio button to turn off the continuous mode.
e. Click on the "Trig" tab to change the trigger settings. The window looks like this:

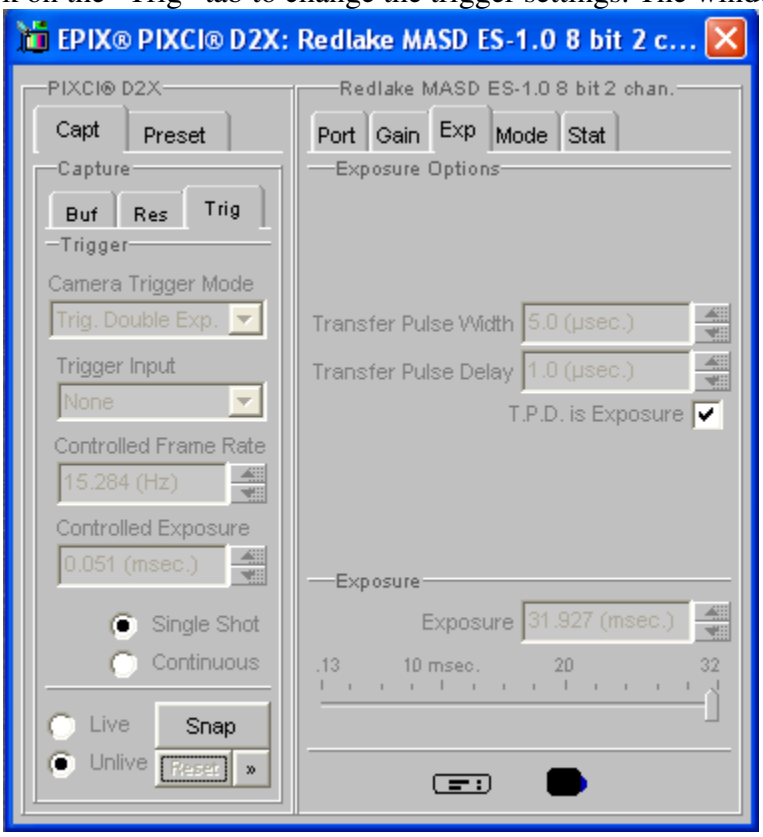

Figure 9 - The XCAP Trigger Screen

f. Set the camera trigger mode to "Trig. Double Exp."
g. Set the trigger input to "Snap Button." If this button is clicked while the pulse generator is running, the camera will take an image on the next incoming pulse.
h. Leave the transfer pulse width at 5.0 µs.
i. Check the "T.P.D. is Exposure" box.
j. Choose an appropriate exposure time (more time will make each image brighter).

4. Pulse generator preparation.
   a. Adjust the settings on the pulse generator to match those listed in **Table 2**.
   b. Press the "Run" button (or equivalent button) to start pulse generation.
5. Particle spreading.
   a. Press the "Start" button on the infusion pump to begin feeding particles onto the water surface. This may take some time as air may be trapped in the syringe needle.
6. Image Collecting.
   a. When ready, click "Snap" in the above window to collect the first pair of images.
   b. Continue to collect images as particles build up on the water surface. If possible, try to capture the advancing film front, if one exists.
   c. Click File→Save Current Buffer to save all the images in the current buffer.
   d. Use image processing software (such as DaVis) to analyze the images.

Images and Results

Two sets of images were taken – one with 10 µm SiO_2 particles, and one with 457 nm SiO_2 particles, both functionalized. The equipment and procedure described above was used to collect all of the images. Noteworthy sets of raw image pairs are shown below.

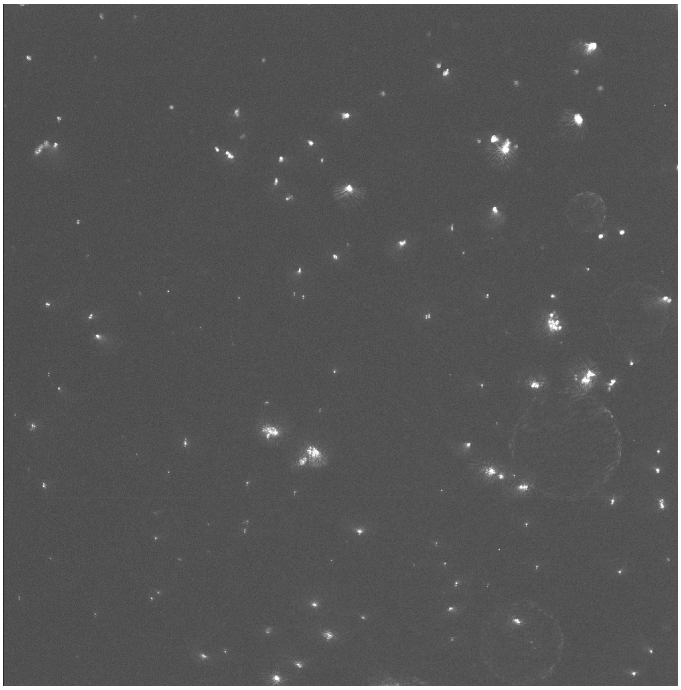

Figure 10 - PIV_SiO2_10um00

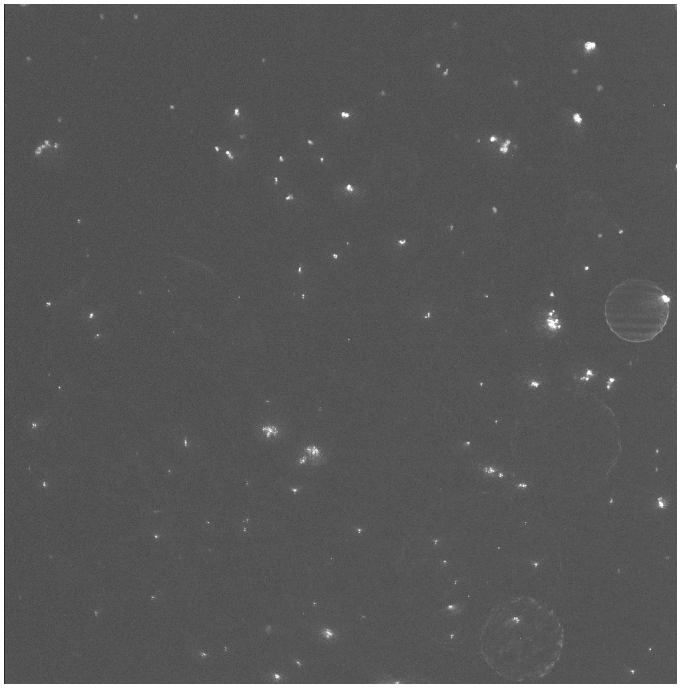

Figure 11 - PIV_SiO2_10um01

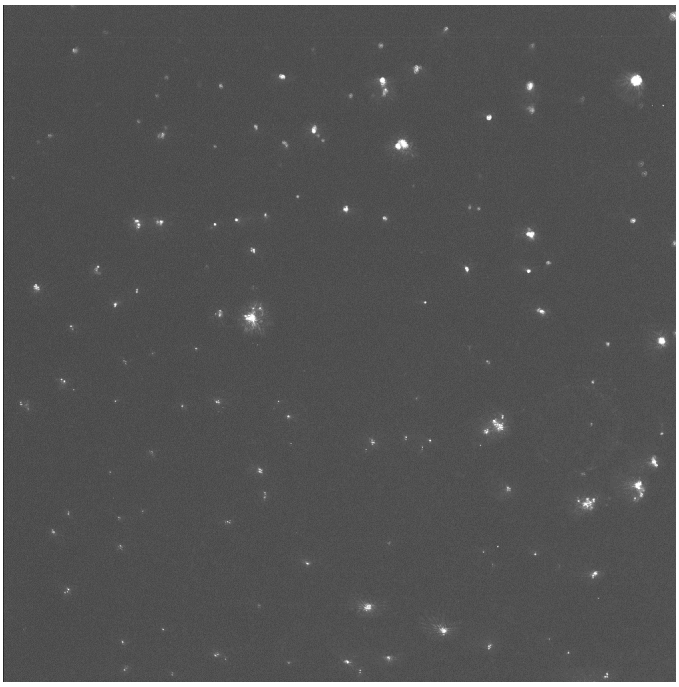

Figure 12 - PIV_SiO2_10um02

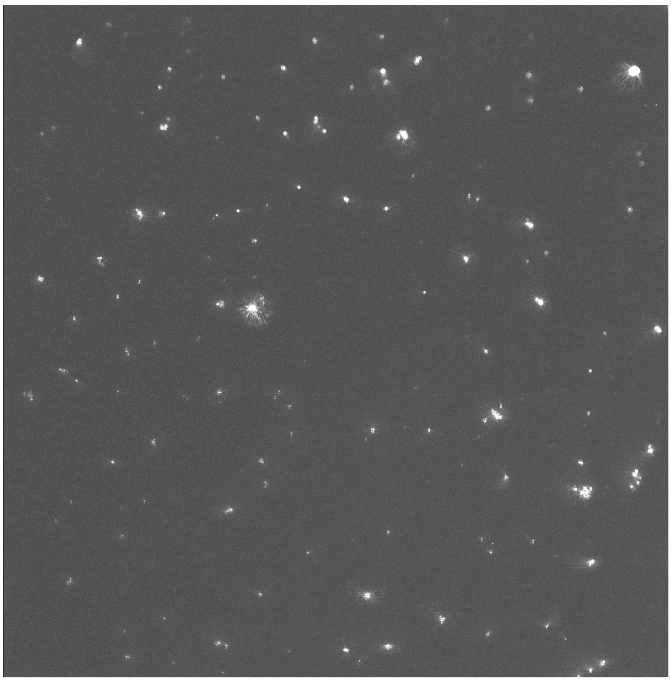

Figure 13 - PIV_SiO2_10um03

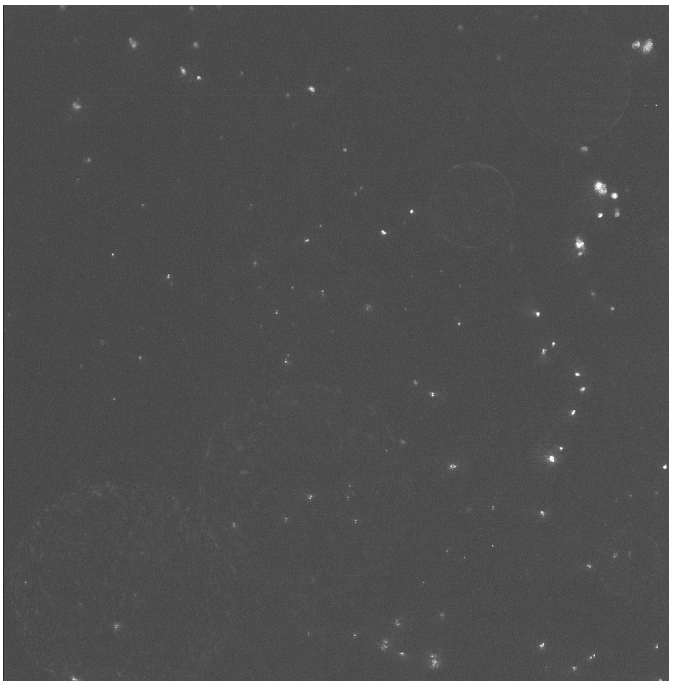

Figure 14 - PIV_SiO2_10um04

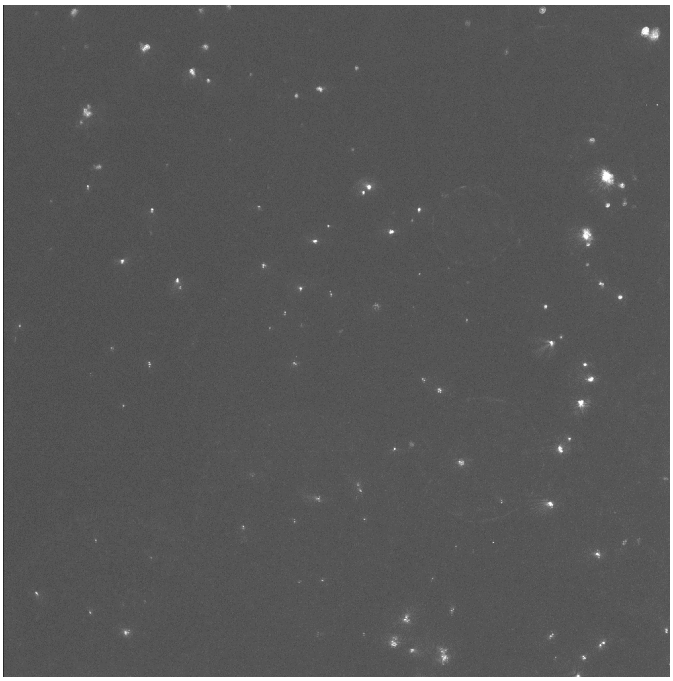

Figure 15 - PIV_SiO2_10um05

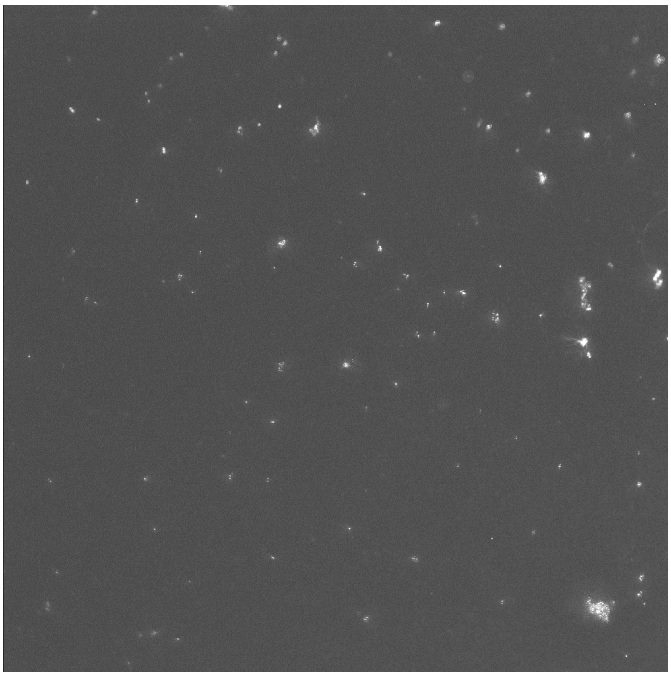

Figure 16 - PIV_SiO2_10um06

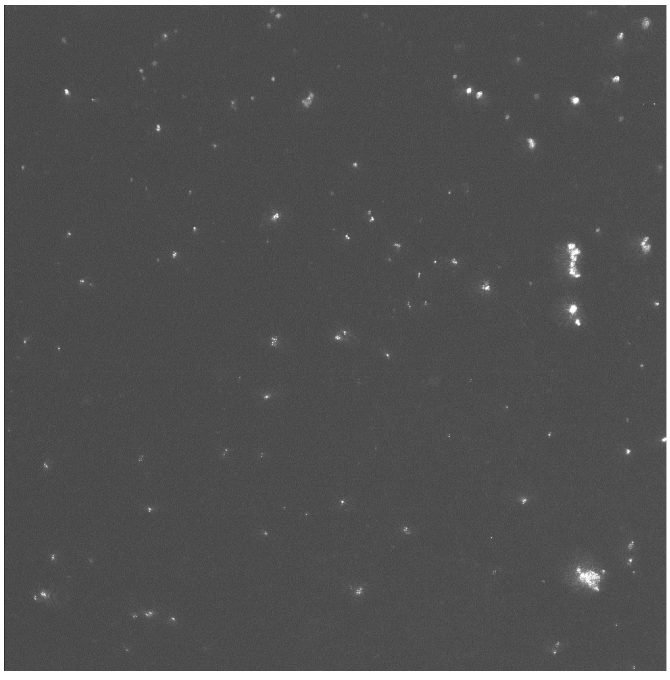

Figure 17 - PIV_SiO2_10um07

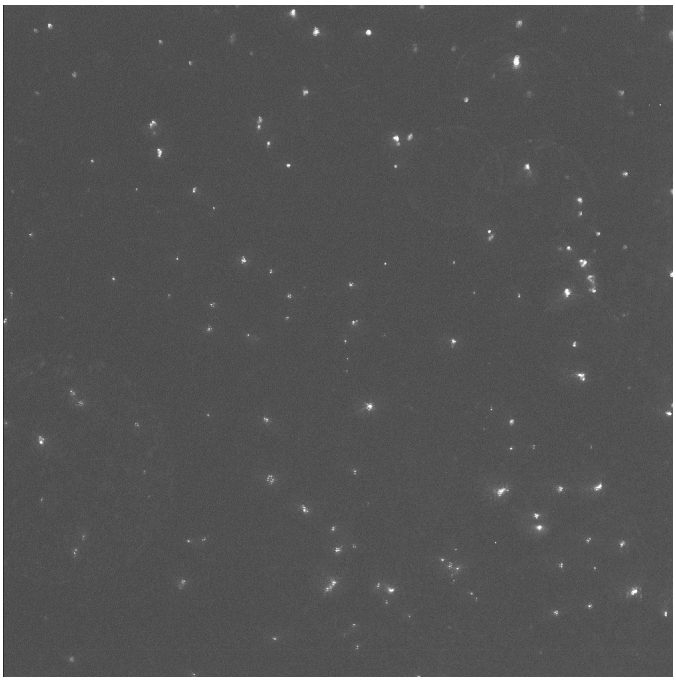

Figure 18 - PIV_SiO2_10um08

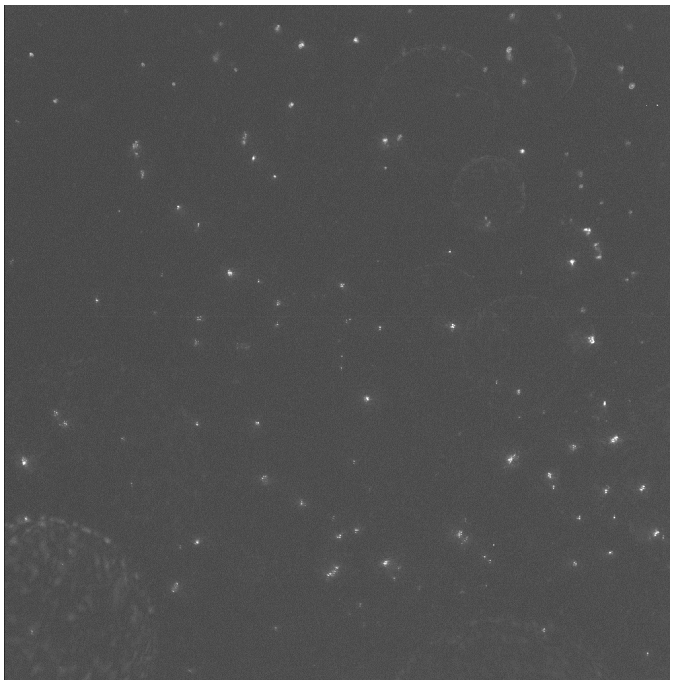

Figure 19 - PIV_SiO2_10um09

Figure 20 - PIV_SiO2_10um12

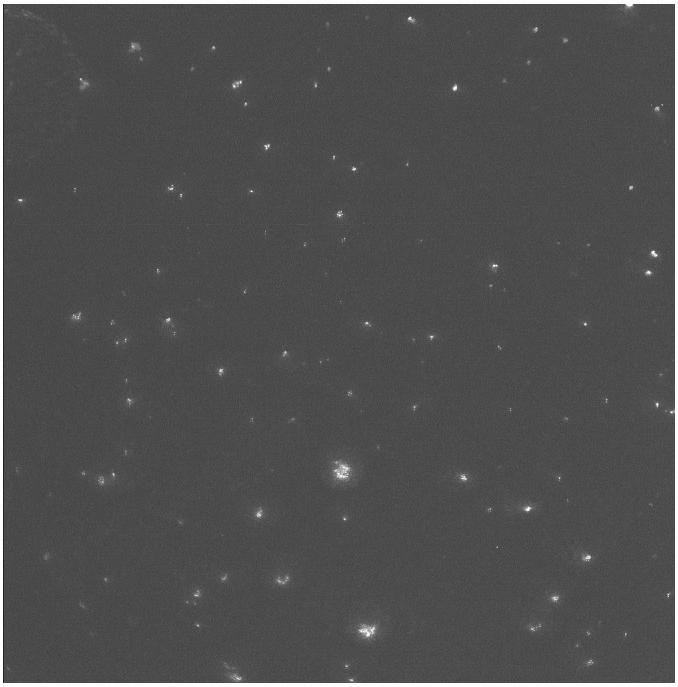

Figure 21 - PIV_SiO2_10um13

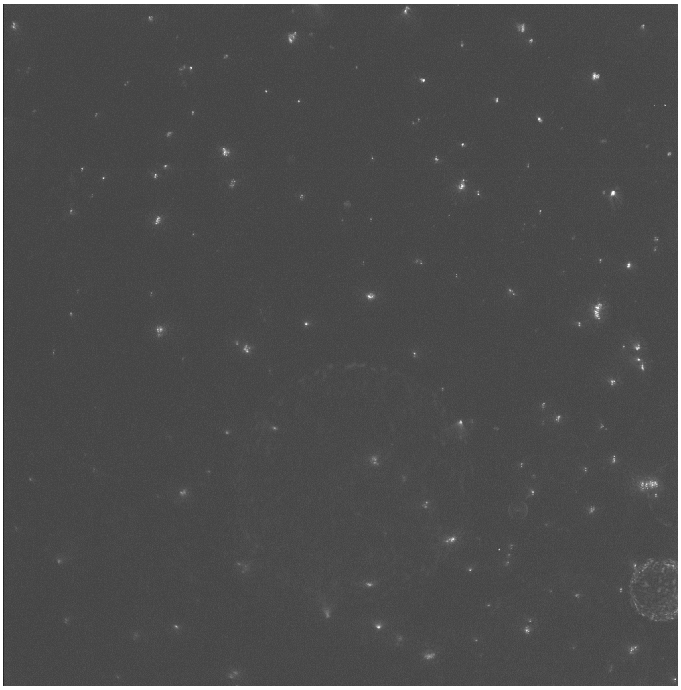

Figure 22 - PIV_SiO2_10um14

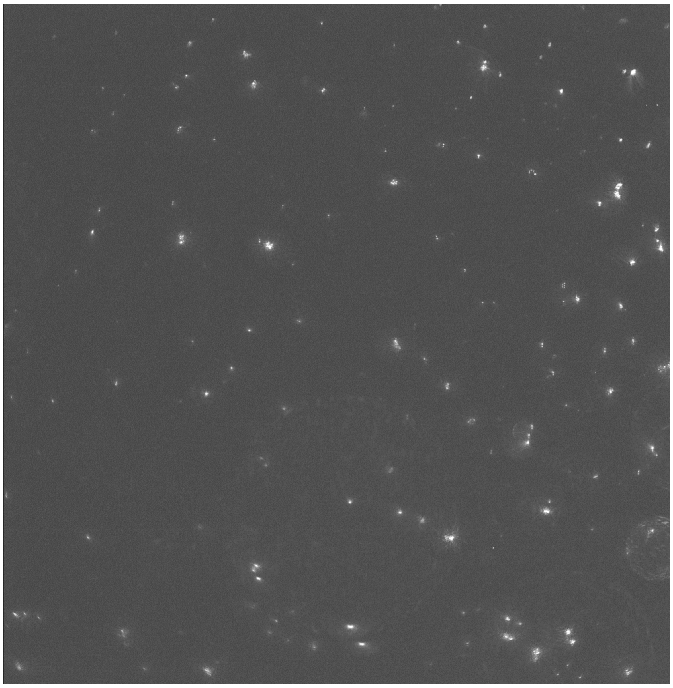

Figure 23 - PIV_SiO2_10um15

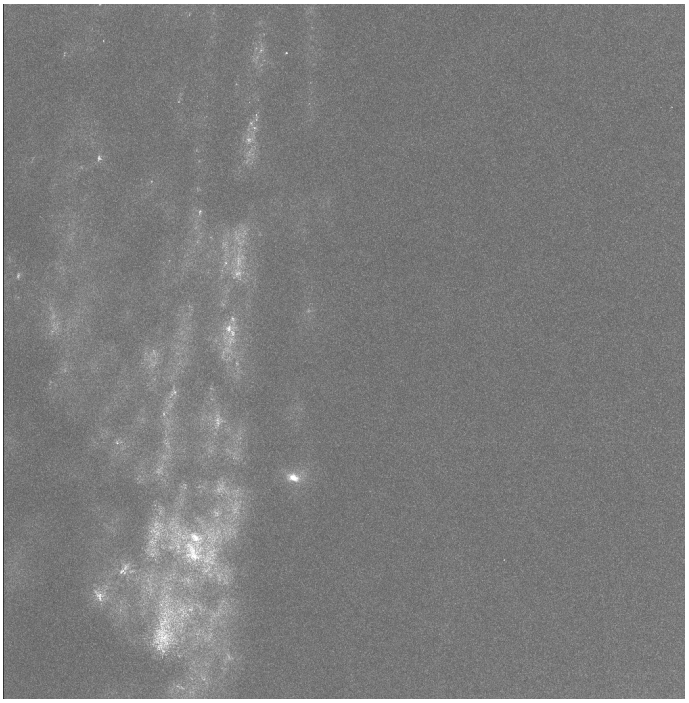

Figure 24 - PIV_SiO2_457nm04

As can be seen from the image directly to the left, the 457 nm particles produced much lower quality images in terms of individual particle resolution. The image shown at the left appears to be an advancing film front, but this is questionable since the other image in the pair (not included) is very different. Since the 10 µm particles produced much crisper images, the analyses were conducted on these image pairs.

Unfortunately, a clear image of an object with features of known dimensions (such as a ruler) could not be obtained with the camera maintained in the experimental position. Luckily, the size scale in the images can still be deduced from the size in pixels of a single, in focus particle in the images. The average particle diameter, judged from multiple images, is about 4 pixels. Therefore, one pixel measures about 2.5 µm of distance.

Each image measures 1000 by 1000 pixels, making the total image about 2.5 mm on each side. By measuring the distance travelled by a single particle and dividing by the interval of time between the two image exposures (or more specifically the time between laser pulses, as these are the only times the particles are illuminated), the velocity of individual particles may be calculated. An example velocity calculation is shown below.

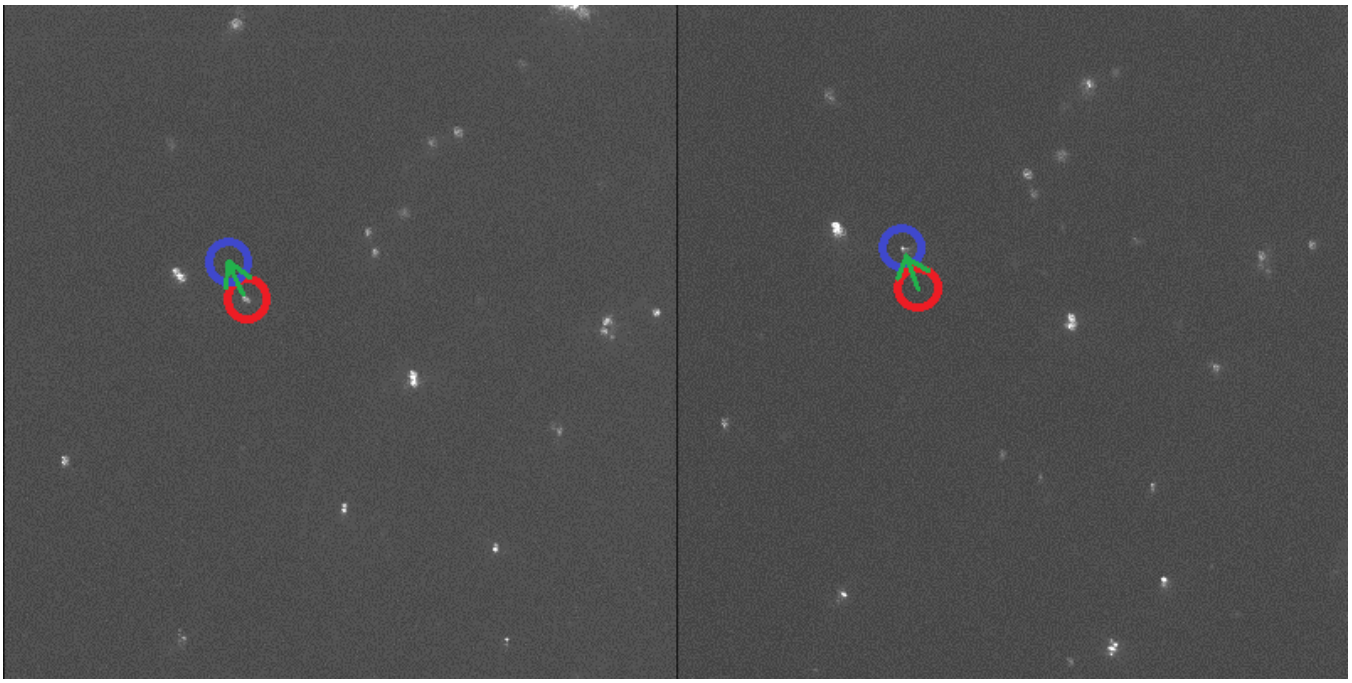

Figure 25 – movement of a single particle in two consecutive images (close-ups of **Figures 16** and **17**)

In **Figure 25**, the top left corner of each image portion can be taken as the pixel location (0,0), with the first number in the pair corresponding to the horizontal difference from the top left corner, and the second number corresponding to the vertical distance from the top left corner. In the image portion on the left, the particle (encircled in red) is located at (144, 175) pixels. In the image portion on the right, the particle (now encircled in blue) is located at (134, 144) pixels. In microns, these locations can be expressed as (360, 437.5) µm and (335, 360) µm. The x and y velocity components of the particle can be calculated by dividing the distance travelled by the time interval (which is known to be 82.5 ms – 49.5 ms = 33ms). Therefore:

$$v_x = \frac{dx}{dt} = \frac{335um - 360um}{33 * 10^{-3} s} = 0.758 mm/s$$

$$v_y = \frac{dy}{dt} = \frac{360um - 437.5um}{33 * 10^{-3} s} = 2.348 mm/s$$

The total magnitude of the velocity is therefore:

$$|\mathbf{v}| = \sqrt{{v_x}^2 + {v_y}^2} = \sqrt{(0.758 mm/s)^2 + (2.348 mm/s)^2}$$

$$= 2.467 mm/s$$

Image Analysis Using DaVis 6.0

In addition, specialized image analysis software may be used to calculate a vector field for an image pair. The software available for this research was DaVis FlowMaster 6.0[7] from LaVision, a utility that allows for vector calculations of image pairs that can be displayed in a visual format.

DaVis has a cross correlation mode that calculates a vector field from an image file composed of two vertically stacked individual images. In order to create an image file, the students in Dr. Tse's group have created a MATLAB program that automatically combined image pairs into a single vertically stacked image. This program's executable is entitled "comboimage" and can be found on the computer near the laser remote controllers in the D-107 laboratory. One of the output images from this program is shown in **Figure 26**.

The procedure from obtaining velocity fields using the DaVis software package is listed separated from the above procedure because these steps are not necessary to perform PIV experiments. However, they are extremely useful to obtain a qualitative visual analysis of what is happening in any given image pair.

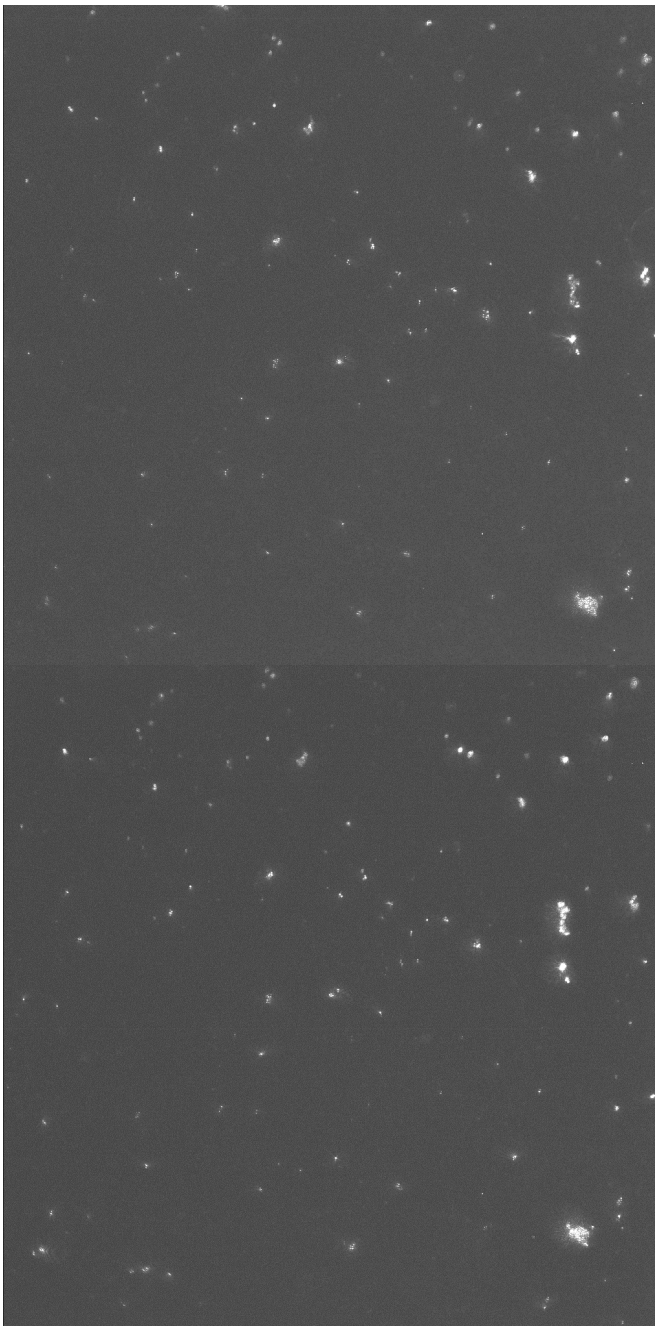

Figure 26 – example image combination used by DaVis to calculate vector fields

Procedure for Obtaining Vector Displacement Fields

1. Open the DaVis software. The initial screen should look something like this:

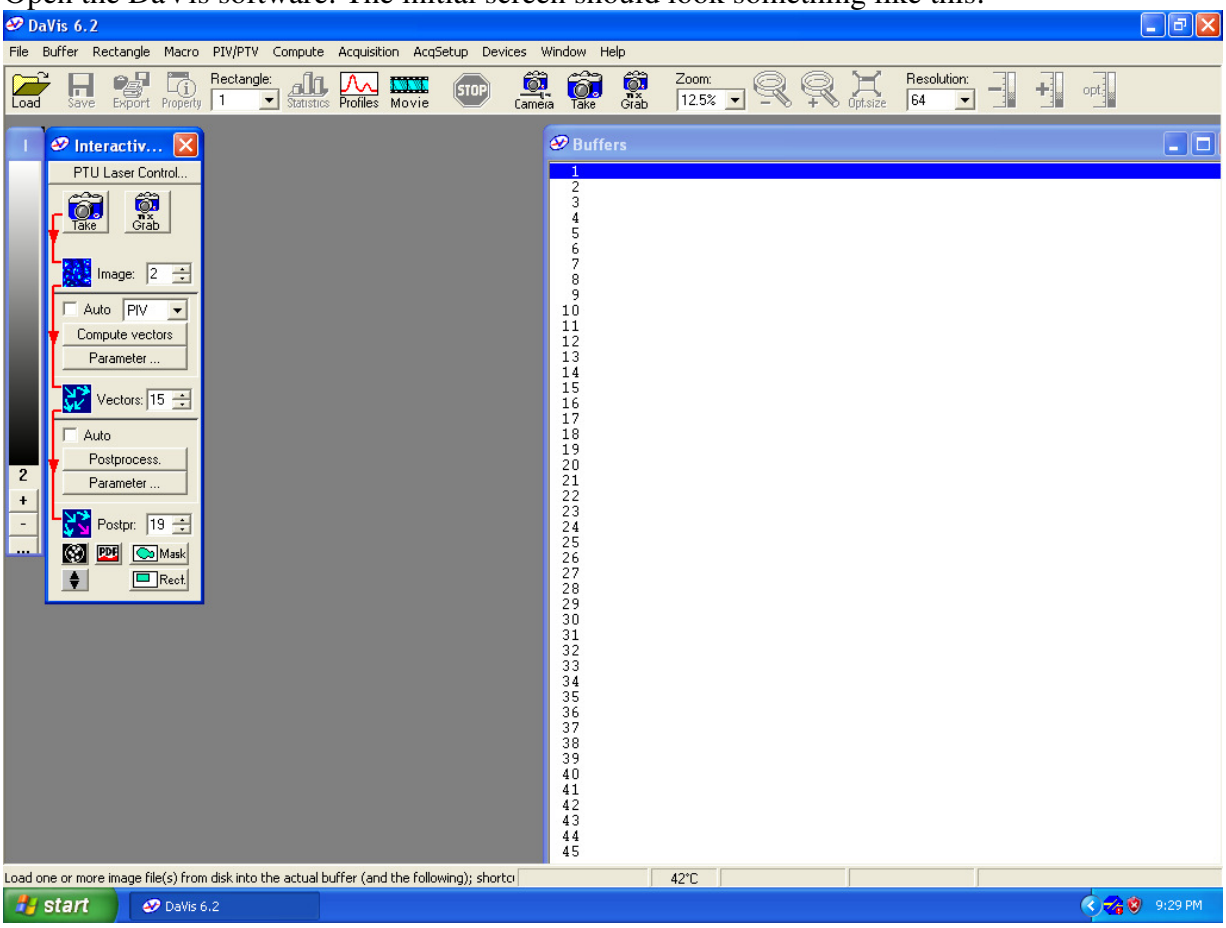
DaVis 6.2
File Buffer Rectangle Macro PIV/PTV Compute Acquisition AcqSetup Devices Window Help
Load Save Export Property Rectangle: 1 Statistics Profiles Movie STOP Camera Take Grab Zoom: 12.5% Opt.size Resolution: 64 opt
Interactiv...
PTU Laser Control...
Take Grab
Image: 2
Auto PIV
Compute vectors
Parameter ...
Vectors: 15
Auto
Postprocess.
Parameter ...
Postpr: 19
PDF Mask
Rect
Buffers
1 2 3 4 5 6 7 8 9 10 11 12 13 14 15 16 17 18 19 20 21 22 23 24 25 26 27 28 29 30 31 32 33 34 35 36 37 38 39 40 41 42 43 44 45
Load one or more image file(s) from disk into the actual buffer (and the following); shortcu
42°C
start
DaVis 6.2
9:29 PM

Figure 27 – DaVis opening screen & interactive window

The window on the left is called the interactive window, and is where the majority of manipulation of the program takes place. On the right is the buffer window, in which input images are loaded by the user and generated images are placed by the program.

2. Load a combined image into the chosen buffer by clicking the Load button.
    a. Make sure the number in the text box next to the "Image" graphic in the interactive window matches the buffer number in which the combined image is loaded.
3. Click the Parameter button in the interactive window. The window featured in **Figure 28** should appear. This is where many of the vector calculation settings can be modified.

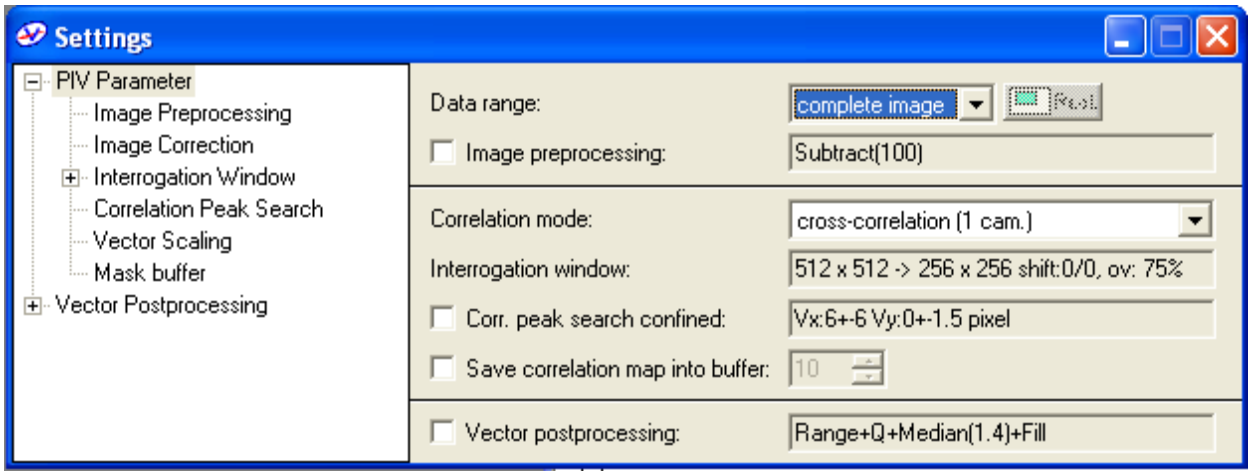

Figure 28 – DaVis Settings window

a. Select the appropriate data range (usually complete image if a vector field for the entire image is desired).
b. Choose the cross-correlation mode (works for two vertically stacked images).

4. Click the Image Preprocessing menu item in the Settings window. This window should appear:

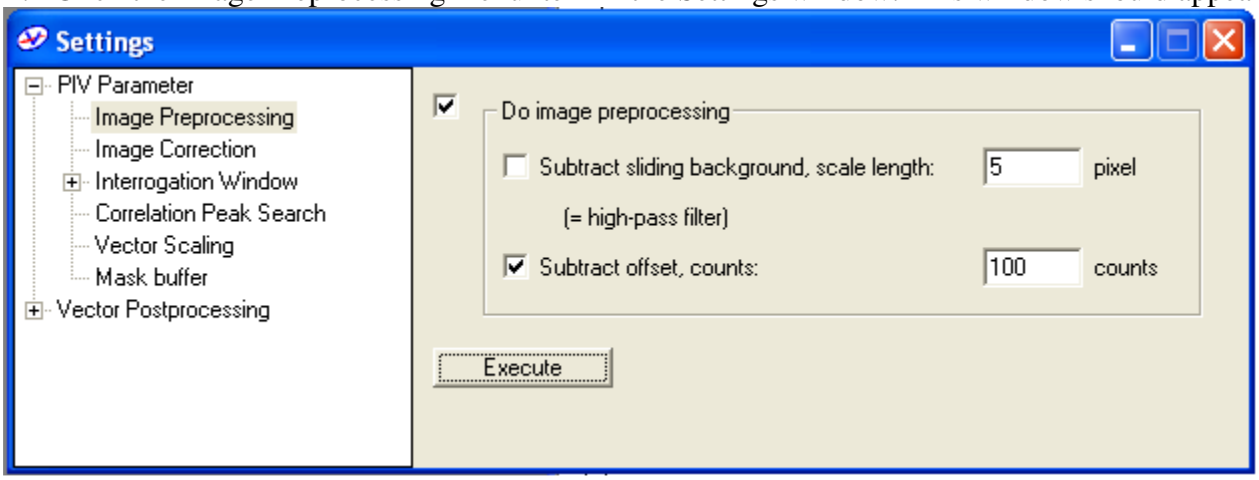

Figure 29 – DaVis image preprocessing window

a. Image preprocessing can be performed to increase the particle/background contrast. Particularly, the preprocessing techniques include a high-pass filter to subtract a sliding background, and an offset subtract that will reduce the intensity of all pixels in the image.
b. If the particle contrast is low, it may be useful to subtract the background intensity in the image. To do this, hover the mouse over background areas of the image to find an approximation of the average background value, and subtract this offset.
c. Click the Execute button to perform the selected image preprocessing. The preprocessed image will appear in a buffer that is selected determined via a text entry in a pop-up.
d. Note: image preprocessing should not be necessary in most experiments.

5. Click on the Interrogation Window menu item. The following window should appear:

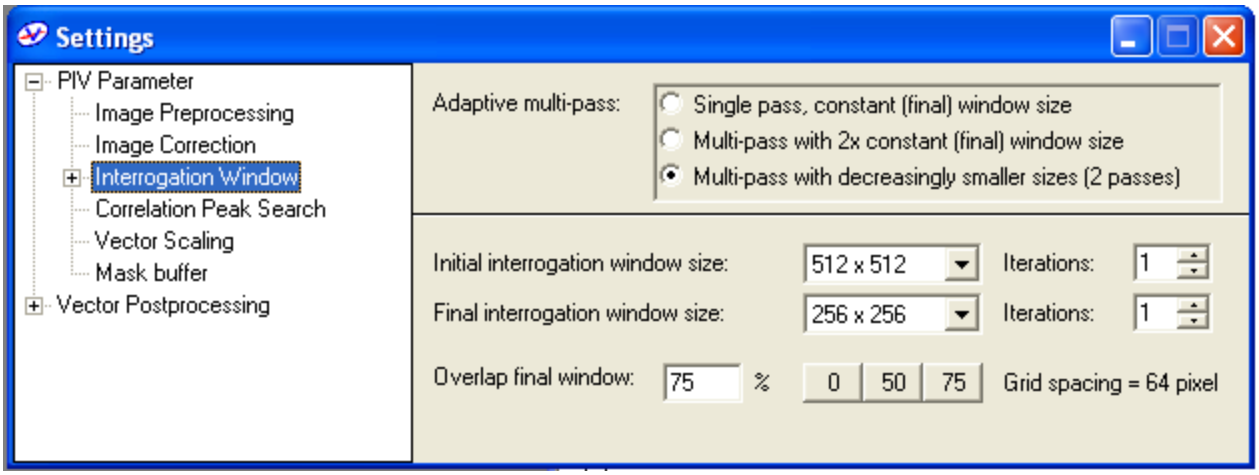

Figure 30 – the DaVis interrogation window screen

a. The settings chosen here affect the actual vector calculation. The algorithm can be chosen as single pass, multi-pass with constant window size, or multi-pass with decreasingly smaller sizes. Usually the latter option is preferable since it will have the program calculate vectors across different size scales, giving a better representation on the whole.
b. The initial and final interrogation window size are the number of pixels that are analyzed by each pass. Vectors are calculated within each interrogation window, and summed at the end of the calculation. These calculations are very processor-intensive and take considerable time to compute; as such, large interrogation window sizes should be chosen when trying to get a vector field for a large image.
c. The overlap of the final window determines how much different interrogation windows in the final pass overlap. Without overlap, a particle that moves from one interrogation window to the next will not be detected as moving. 75% overlap is usually sufficient.

6. Choose the appropriate buffer for the input data (i.e. preprocessed or not).
7. Select the appropriate buffer for the vector field before it is calculated.
8. Click the Compute Vectors button to begin vector calculation. The process may take up to 5 minutes to complete.
9. Computed vector fields may be saved in various image formats using File→Export.

Vector Displacement Fields of Select Image Pairs

Below are shown some of the vector fields calculated using DaVis from the particle images shown above. Note that the flow direction is not uniform in all the image pairs – this is because most of the images were taken in the time period before a film front began to materialize, and as such, the particle motion was essentially random over large time scales. In fact, in this set of experiments (only 10 µm particles could be studied), a consistent film front failed to develop, most likely because the particles are simply too large to self-assemble into neat hexagonal grids. However, clear patterns in particle velocities can be seen in some of the images, indicating that over small time scales, the behavior is more uniform.

Also note that even over very short time scales, the vector displacement fields need not be uniform in direction. In many cases, the field may exhibit curvature (as seen in **Figure 33**), or even vast changes in magnitude in the small area imaged (as seen in **Figure 35**).

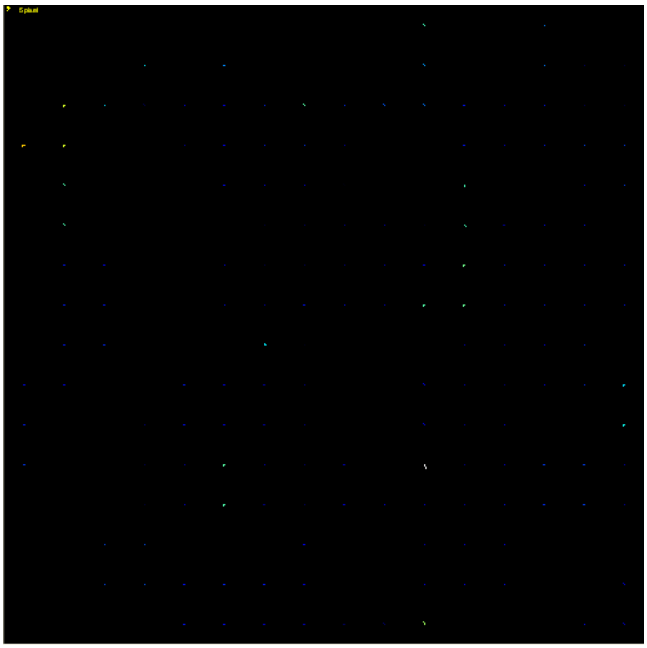

Figure 31 – PIV_SiO2_10um_0001. Control images taken with the system essentially at rest.

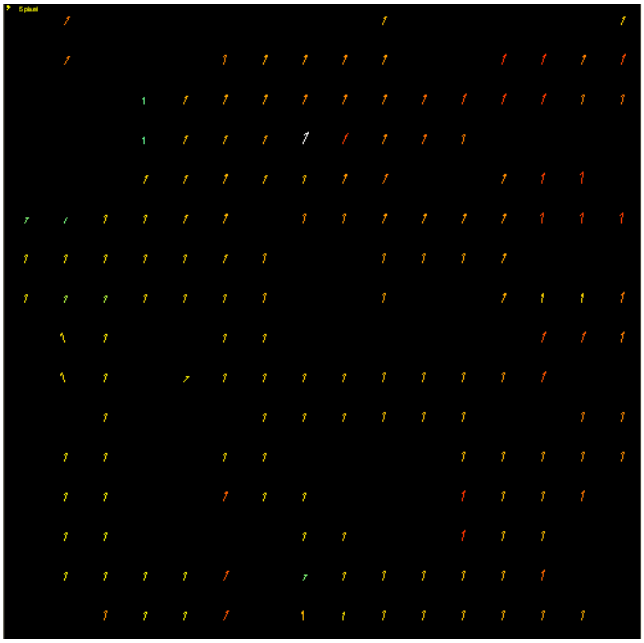

Figure 32 – PIV_SiO2_10um_0203. Slight displacement towards the top right of image.

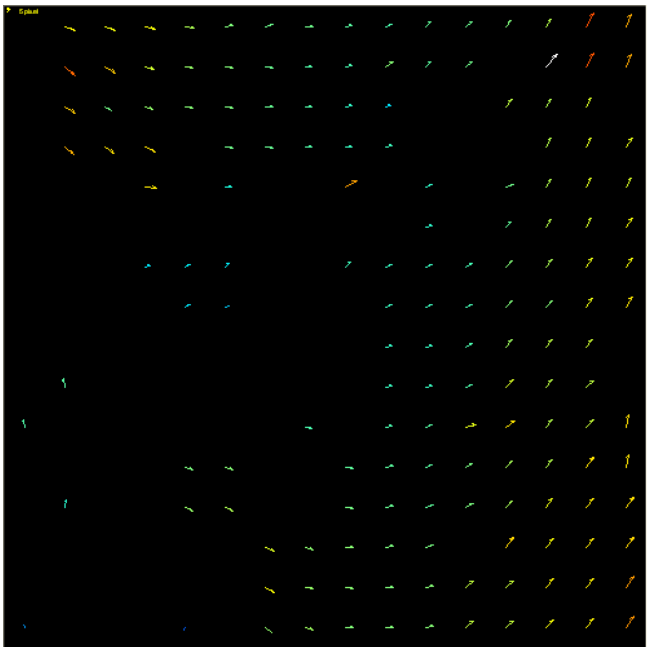

Figure 33 – PIV_SiO2_10um_0405. Vector field shows an interesting curved geometry.

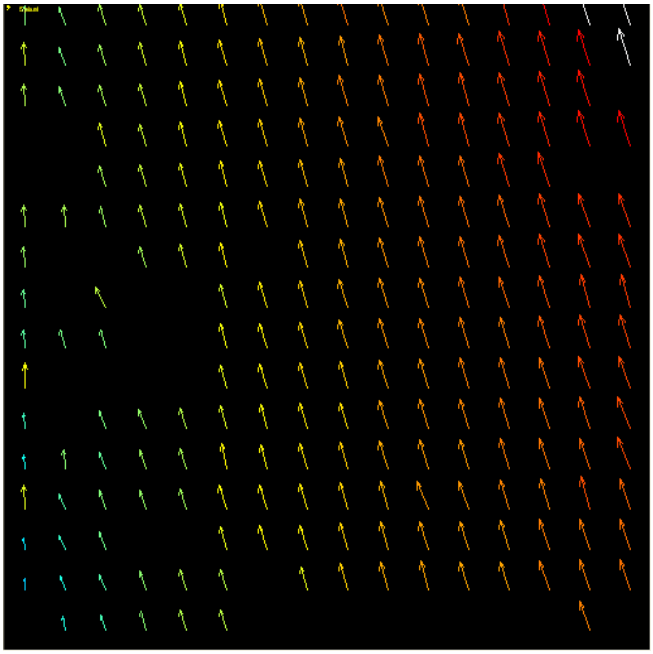

Figure 34 – PIV_SiO2_10um_0607. Shows fast, uniform movement towards top left.

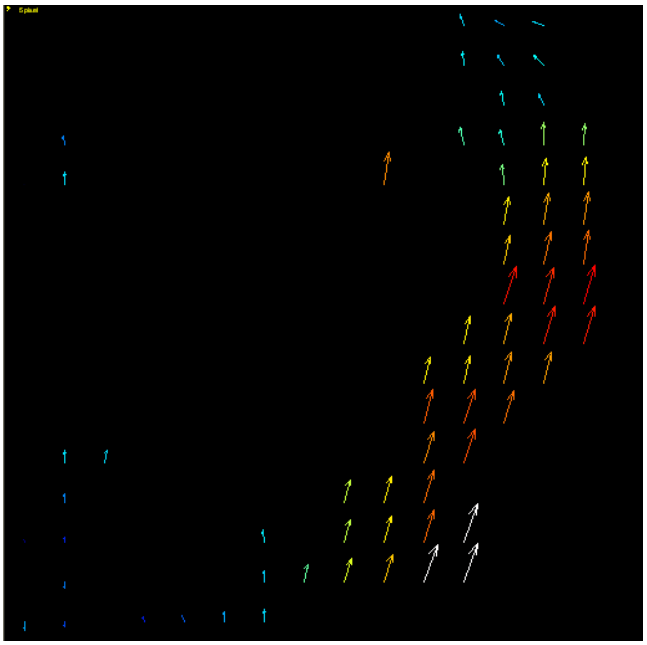

Figure 35 – PIV_SiO2_10um_0809. Shows displacement some areas but not others.

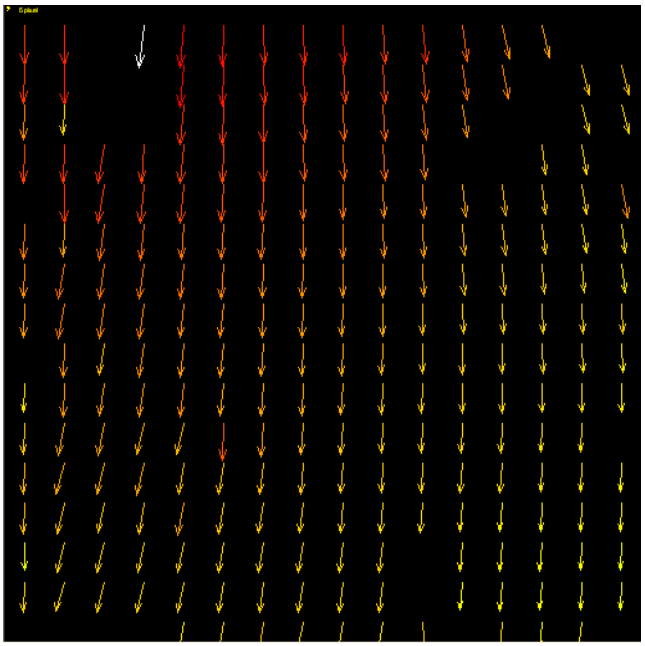

Figure 36 – PIV_SiO2_10um_1213. Shows fast, uniform downward displacement.

To obtain the data set describing the above vector fields from DaVis, simply select File→Export Buffer→Spreadsheet into Clipboard, as shown in **Figure 37**.

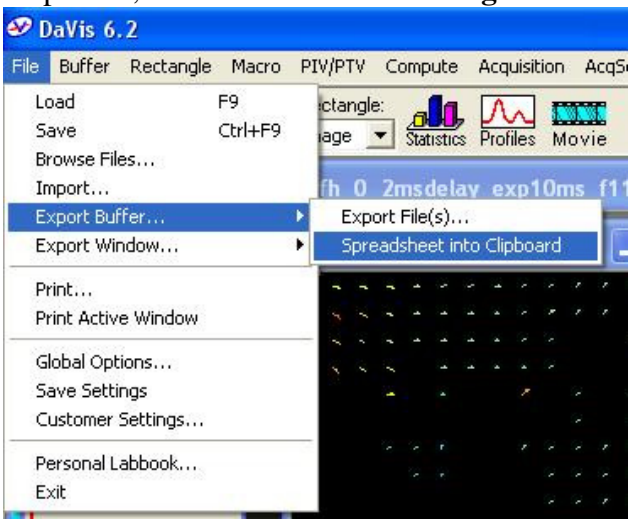

Figure 37 – how to export DaVis vector data into a spreadsheet.

Once this done, the data may be directly pasted into an open Excel spreadsheet. **Table 3** shows the data set for PIV_SiO2_10um_0405 (minus the header columns):

x	y	vx	vy
32	32	0	0
96	32	16.8389	6.51544
160	32	16.5529	6.02293
224	32	16.7619	5.62245
288	32	14.6966	3.19016
352	32	13.5553	-0.06003
416	32	12.3802	-3.29613
480	32	12.8949	1.10971
544	32	11.9815	-1.30721
608	32	11.0043	-3.82608
672	32	9.86491	-7.60171
736	32	10.0736	-7.85688
800	32	9.42592	-9.70672
864	32	9.8911	-11.6444
928	32	13.075	-20.2813
992	32	9.86122	-18.3771
32	96	0	0
96	96	17.3767	15.4833
160	96	16.8825	10.1818
224	96	15.2993	5.63021

Table 3 – exported DaVis vector data

These are only the first few rows of the spreadsheet. The "x" and "y" columns refer to the x and y pixel position of the image, while the "vx" and "vy" columns refer to the x and y

components of the velocity at that particular location, measured in pixels per time difference between image exposure. If the size scale of the image as well as the time between exposures is known, these velocity components can be easily calculated in meters per second.

Discussion

The most pressing problem facing these experiments is the inability of large (10 μm) particles to self-assemble neatly under current functionalization conditions. There are a few remedies that may be attempted to correct this issue. Functionalization studies may be conducted on large particles to identify better functionalizing agents/surface concentrations that yield densely packed particle films. In addition, smaller particles may be used with known, working functionalization conditions, but the particles have to be large enough to be seen with the camera. As was roughly calculated above, each pixel in the images investigated corresponds to roughly 2.5 μm, so this should be the smallest particle size that can still be seen directly by the camera. A study of particle visibility vs. particle size should be conducted to determine the smallest feasible particle size that can be used. Finally, other possibilities to make the particles more visible should also be investigated. For example, fluorescent dyes may be incorporated into the particles to make them release wavelengths of light that can be picked up by the camera. If this is done, it may be possible to see individual particles smaller than the resolution of the camera. Indeed, this may be already possible, since even very small particles will exhibit Rayleigh scattering to some degree that may be observed. However, as seen from the image of 457 nm particles in **Figure 24**, it is most likely that individual, small particles will be very difficult to see when the film is densely packed.

Also, it should be noted that the current experiments are limited by the hardware available. Better lenses (or indeed better cameras) may be used to clearly observe individual particles smaller than 2.5 μm. In addition, the wavelength of laser light used may be changed to a smaller wavelength to better image smaller particles. However, regardless of hardware, it will be basically impossible to decrease the optical resolution limit below about 200 nm.[8] In any case, it should be possible to clearly image closely-spaced particles smaller than 1 μm optically, and this possibility should be investigated.

Finally, it should be noted that the composition selections in these experiments were somewhat arbitrary. Silica particles were used simply for ease of synthesis and availability. Octadecyltriethoxysilane was used because it was previously demonstrated to provide good film density, but an in-depth study of film quality vs. functionalizing agent has yet to be conducted. Finally, isopropanol was used because of its high spreading tension, but there may be other spreading agents that are able to create particle films more quickly and uniformly. There are many different chemical combinations that may yield better results than the ones investigated here, and though theoretical considerations can suggest combinations that are more likely to be effective, in the end, the "best" combinations (which vary according to what effect is desired) must be determined experimentally.

Conclusion

This report shows a proof of concept of particle imaging velocimetry of 10 μm functionalized silica particles. Calculation of vector displacement fields using specialized software has also been demonstrated. However, many aspects of this realm of experimentation are left to be investigated. In particular, the effect of variations in the size of particles used and the chemistry of the system components have yet to be studied. Regardless, a working experimental framework has been demonstrated that may be adapted to yield better results in the future. Hopefully, future experiments will determine a generalized fluid forming method

that can be used with many different chemistries to create densely packed thin particle films, effectively transfer those films onto a large range of useful surfaces, and successfully incorporate them into a variety of technical applications. If these goals can be accomplished, these methods may be shown to be a viable alternative to conventional thin film processing techniques.

References

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